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RESEARCH OF ACOUSTIC EMISSIONS IN NATURAL GAS VEHICLE STORAGE CYLINDERS

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17. Abstract

A detailed report on a project aimed at validating Acoustic Emission Testing (AET) as a means of detecting active cracking in compressed natural gas vehicle storage cylinders.

The report includes the procedures followed, the results obtained, and the final conclusions drawn from testing AET.

Results indicated that acoustic emission (AE) testing was able to detect stress corrosion cracking, with activity levels very sensitive to cylinder pressure. However, AE may not be able to predict imminent burst.

Testing of a larger number of cylinder samples is recommended.

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Brampton (Ontario)
L6T 1A7

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17. Résumé

Rapport détaillé sur le projet visant à faire reconnaître le dépistage des émissions acoustiques (DEA) en tant que moyen de déceler le fêlage ou la fissuration des réservoirs de stockage cylindriques des véhicules au gaz naturel comprimé.

Le rapport comprend les modalités suivies, les résultats obtenus, et les conclusions définitives au sujet du DEA.

Les résultats ont indiqué que le dépistage des émissions acoustiques (EA) pouvait déceler la fissuration par corrosion sous tension, avec niveaux d'activité très sensibles à la pression des réservoirs cylindriques. Toutefois, les émissions acoustiques peuvent ne pas prédire une explosion imminente.

On recommande le dépistage des émissions acoustiques sur un plus grand échantillonnage de réservoirs cylindriques.

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RESEARCH OF ACOUSTIC EMISSIONS
IN NATURAL GAS VEHICLE
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Scientific Authority = Mr. Pat Hallet
(613) 991-6043

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Contact Administered by:
Science & Professional Services Directorate
Supply and Services Canada
11 Laurier Street - 12C1
Hull, Quebec K1A 0S5
Contact: Mr. Michel Rancourt

Report submitted by:
CNG Fuel Systems
2150 Steeles Avenue, East
Brampton, Ontario L6T 1A7
Contact: Stephen A. Carter

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EXECUTIVE SUMMARY

The use of compressed natural gas (NGV) as an auto fuel typically involves the on-board storage of NGV at 3000 PSIG in thick walled high strength steel cylinders.

A cylinder pressurized to 3000 PSIG contains an enormous amount of energy. Should such a cylinder rupture, the energy unleashed from the NGV could propel cylinder fragments in a bomb-like fashion. Accordingly, regulations require the periodic requalification of in-use NGV cylinders to minimize user risk.

The currently used retest method is a hydro test. That test over-pressures the cylinder with water and measures both leakage rate and permanent expansion of the cylinder. The hydro test is undesirable from two standpoints:

- It over-pressures the cylinder (reduces cylinder strength, life)
- It requires that the cylinder be removed from the car (expensive)

Acoustic Emission Testing (AET) has been proposed as an alternative to the hydro test. AET is based on the theory that a crack in a cylinder emits a characteristic acoustic signature which can be transduced and recognized. An AET monitoring system would acoustically monitor a cylinder during filling and would reject the cylinder if it recognized the crack signature.

The factors in favor of AET as an alternative test method include:

- Doesn't require cylinder removal from car
- Doesn't over-pressure the cylinder
- Could be performed every time the vehicle is filled

A project funded by the Ontario Ministry of Energy showed AET to be promising as an alternative to the hydro test. However, that project primarily dealt with cylinder sections, leaving concern that the behavior of complete cylinders might be different. The report from that project dovetails nicely with this work to provide a more complete view of AET and is recommended reading.

This project was aimed at the concern about complete cylinder behavior. The goal was to induce severe, accelerated corrosion and to monitor the cylinder's acoustic emissions from initial pressurization until the cylinder fractured.

NOTE: This destructive test was aimed solely at validating AET as means of detecting active cracking. There are no conclusions relative to cylinder life expectancy in normal service.

The test was performed outdoors at CFB Borden. The cylinder did corrode, crack, and burst. Thus, active cracking was created by the corrosive charge. Acoustic emissions were monitored during the period from initial charge until burst, and the following conclusions are drawn from the results:

- Cylinders are acoustically active during & after filling
- AET appears able to recognize the presence of a crack
- AET may not be able to predict remaining life before burst
- AET may not be able to predict imminent failure
- Further work with a larger sample size is indicated

SOMMAIRE

L'utilisation du gaz naturel comprimé (GNC) pour propulser les véhicules automobiles comporte en général le stockage à bord de ces derniers, dans des réservoirs cylindriques en acier très résistants et à parois épaisses, et à une pression manométrique de 3 000 lb/po^2.

Un réservoir où la pression manométrique est de 3 000 lb/po^2 contient une quantité énorme d'énergie. Si jamais il se brisait, l'énergie produite par le GNC risquerait d'en projeter les fragments à la façon d'une bombe. C'est pourquoi la réglementation exige l'examen périodique des réservoirs de GNC en usage, pour réduire au minimum les risques courus par les utilisateurs.

Le mode de vérification utilisé actuellement est un essai hydraulique permettant de surpressuriser hydrauliquement les réservoirs et d'en mesurer tant le taux de fuite que l'expansion permanente. Cet essai n'est pas souhaitable, pour deux raisons:

- Il surpressurise les réservoirs (ce qui en réduit la résistance, la vie utile).
- Il oblige à enlever les réservoirs de la voiture (ce qui est coûteux).

Le dépistage des émissions acoustiques (DEA) a été proposé comme solution de rechange à l'essai hydraulique. Il est fondé sur la théorie selon laquelle une fissure ou un fêlure qui se produit dans un réservoir émet des sons caractéristiques qui peuvent être captés et reconnus. Il consisterait à ausculter le réservoir pendant le remplissage et permettrait de l'éliminer si on décelait les signes acoustiques d'une fissure ou d'une fêlure.

Les facteurs qui militent en faveur du DEA en tant que méthode d'essai sont les suivants:

- Il n'oblige pas à enlever les réservoirs de la voiture.
- Il n'oblige pas à surpressuriser les réservoirs.
- On peut l'effectuer chaque fois qu'on fait le plein du véhicule.

L'exécution d'un projet financé par le ministère de l'Énergie de l'Ontario a montré que le DEA pouvait être une solution de rechange avantageuse à l'essai hydraulique. Toutefois, ce projet a porté surtout sur certaines parties des réservoirs, ce qui donne à penser que le comportement de l'ensemble de ces derniers pourrait être différent. Le rapport afférent s'ajoute avantageusement au document dont il est question ici: il donne une meilleure idée du DEA, de sorte que nous en recommandons la lecture.

Dans le cas présent, il s'agissait de répondre aux préoccupations au sujet de la résistance ou du comportement de l'ensemble des réservoirs, en provoquant une corrosion grave et accélérée ainsi qu'en décelant les émissions acoustiques desdits réservoirs, depuis la pressurisation initiale jusqu'au bris de ces derniers.

NOTA: Cet essai destructeur n'avait pour but que de faire reconnaître le DEA en tant que moyen de déceler la fissuration ou le fêlage en train de se produire. Il n'a pas permis de tirer des conclusions quant à la durée de vie probable des réservoirs utilisés normalement.

L'essai a été effectué en plein air, à la BFC Borden. Le réservoir visé s'est corrodé et fissuré, puis il a éclaté. La fissuration effective a par conséquent résulté de la charge corrosive. Les émissions acoustiques ont été contrôlées au cours de la période comprise entre la pressurisation initiale et l'éclatement, et les résultats obtenus ont permis de tirer les conclusions suivantes:

- Les réservoirs cylindriques émettent des sons pendant et après le remplissage.
- Le DEA semble permettre de reconnaître la présence d'une fissure ou d'une fêlure.
- Le DEA ne permet peut-être pas de prédire pendant combien de temps les réservoirs vont tenir avant d'éclater.
- Le DEA ne permet peut-être pas de prédire une défaillance imminente.
- Il faut effectuer d'autres travaux portant sur un échantillon plus vaste.

BACKGROUND

OVERALL CONCEPT

The concept's goal was to provide the technology for performing AET on vehicles during the normal filling process. That would permit vehicles to have their cylinders tested during each fill cycle. The presumed test sequence would be:

Vehicle parks at filling site
AET sensor attached to cylinder with a coupling gel
AET system monitors cylinder during & immediately after filling
AET sensor disconnected, system makes pass/fail determination

That overall concept includes the opportunity to centrally computerize fill site AET and thus maintain a broad based tracking system based on the cylinder's: size, registration number, and age; geographical location; and AET performance.

BASIC ASSUMPTIONS

The basic assumptions underlying the AET concept evaluated here are shown below:

- A crack emits acoustic energy during pressure & temperature changes
- The acoustic energy has a recognizeable characteristic signature
- The AET data is clearest during and/or immediately after filling
- No AET data exists on a stabilized cylinder (temperature, pressure)
- Cylinders with cracks are potentially unsafe, and should be retired
- Cracks in cylinders may occur from a number of causes, including:
    * fatigue
    * stress corrosion cracking (SCC), hydrogen embrittlement
    * tooling marks from stamping the blank into a cylindrical shape
    * metal folds from rolling/forming the open end of cylinder shut

PRECEDING WORK

A companion project funded by the Ontario Ministry of Energy preceded this project. That project evaluated AET with the primary focus being on sections of cylinders. The conclusions from that work were that AET has promise, and appears able to recognize cracks when utilized immediately after filling.

However, there were some notable concerns about the similarity of complete cylinders and sections of cylinders. Some of the concerns are summarized below:

Areas of Concern re total cylinder vs cylinder section	Speed of sound in cylinder wall
	Impedance of cylinder wall to acoustic waves
	Transport distances from crack sites to transducer
	Impedance change sites acting to reflect acoustic waves
	Viability of existing AE signal-noise ratio

That preceding work suggested a demonstration field test (test to destruction) to reduce the concerns about testing cylinder sections. The concept was to induce SCC in a virgin cylinder and monitor acoustic emissions during the corrosion period. If the cylinder failed, cracks would obviously have been generated, and the AET systems performance could be validated or invalidated.

PRECEDING WORK [continued]

A number of conclusions were drawn in the companion work funded by the Ontario Ministry of Energy which are releveant here. Those conclusions include the following:

1. Significant, readily detectable acoustic emissions (AE) occur
2. Those AE levels are easily processed with current equipment
3. Useable AE appeared in both complete & sectioned cylinders
4. Future work is deemed best performed on total cylinders

5. Some exposure time to a corrodant is required before AE appear

6. After initially appearing, AE seemed to exist "continuously" until cracking or failure occurred.
   - AE appear quite sensitive to cylinder wall stress, ie AE would disappear as cylinder pressure/stress drops and reappear as cylinder pressure/stress rises

7. Fill-cycle AET may be feasible only after filling stops
   - AE levels are much higher during filling vs afterwards
   - Level of AE sources during fill are not known or ranked
     * crack initiation, crack growth
     * NGV flow induced noises
     * cylinder wall grain-creep re 3-D wall expansion
     * gases entering wall, changing state inside wall
   - AE levels during fill not rigorously evaluated
     * data appeared to be all noise
     * Excessive system gain could have lost good data
     * no useable data saved re further manipulation
   - AE levels after filling appear stable & useable
     * data appears good for several minutes after
     * time window where data valid not determined

8. AET's ability to identify corrosion or cracking mechanisms is unknown (ie recognition of sulphide stress corrosion cracking, crack nucleation, hydrogen embrittlement, fatigue, etc)

RESEARCHERs

This project was proposed following a series of meetings including CNG Fuel Systems, Transport Canada, Viatec Resource Systems, Inc., and the Ontario Ministry of Energy. The test work was conducted with Viatec as the primary researcher, with their primary contact (Mr. Michael McKinnon) located at:

Viatec Resource Systems, Inc.
#114, 6815 8th Street Northeast
Calgary, Alberta T2E 7H7
(403) 291-3137

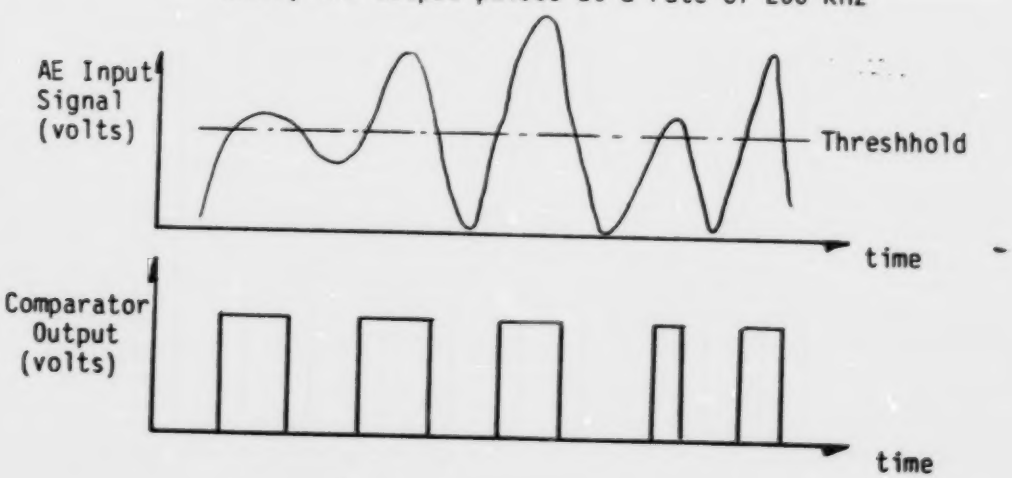
CNG Fuel Systems served as program administrator. If the technology were market ready, CNG Fuel Systems expected to commercialize it in the market.

AET SYSTEM DESCRIPTION & FUNCTIONAL BLOCK DIAGRAM

The AET system concept, as used here, has the following componentry:

A.E. Sensor	responds to acoustic energy (typ measured in microBars) by producing a voltage with similar spectral content
Couplant	a gel-like viscous fluid used to acoustically couple the sensor to the test specimen (ie cylinder wall)
Pre-Amplifier	if required, boosts signal from AE sensor to maintain immunity from electromeagnetic noise interference (EMI) and to facilitate further signal processing
Filter	discards AE signals from frequency ranges where poor signal to noise ratio exists or where no AE data exists
Activity Comparator	compares filtered AE data to pre-programmed threshhold values to determine if instantaneous data is indicating noise or actual acoustic activity. Note: this stage's output is hereafter referred to as AE Counts.

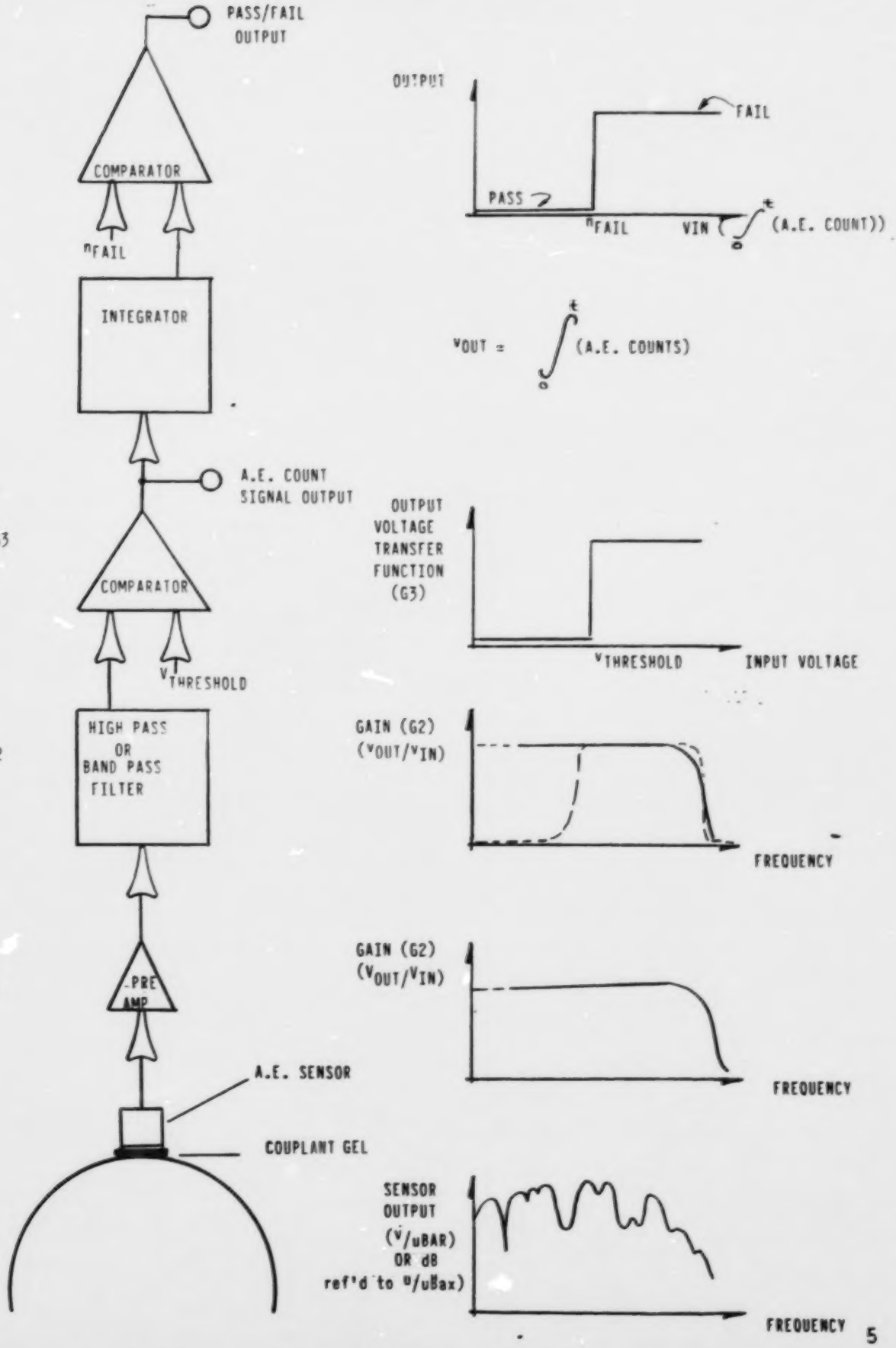
Example: while a 200 KHz signal exceeds the threshhold level, the output pulses at a rate of 200 KHz



Integrator	integrates the signal from the Activity Comparator to totalize the counts during the integration period
Pass-Fail Comparator	compares output of the integrator with pre-programmed values to determine whether cylinder passes or fails

The following block diagram portrays these functional blocks and the format in which component specifications might be offered.

AET SYSTEM BLOCK DIAGRAM



CONCLUSIONS

CONCLUSIONS

The test cylinder corroded, cracked and burst after 190 hours of testing. At burst, the cylinder suffered three longitudinal fractures and actually separated into two separte fragments. Both fragments were flung into the air and landed 140 meters away from the test site. The crack origin point was identified as a 100 mm long brittle fracture zone. The remaining fracture surface areas were indicative of ductile (shear) cracking.

The cylinder was monitored continuously during the 190 hours of testing. While the cylinder was generally active during the period, there was NO indication of increasing activity immediately prior to the cylinder rupturing.

In general, the preliminary conclusions relative to AET that were noted on Page 3 of this report were validated.

1. Significant, readily detectable acoustic emissions (AE) occur
2. Those AE levels are easily processed with current equipment

3. Some exposure time to a corrodant is required before AE appear
   - an incubation time is required for AE to exceed the detection threshhold; perhaps related to corrosion reaction kinetics or crack nucleation mechanisms

4. After appearing, AE activity remained until the cylinder burst
   - once AE activity appeared, it was high after each fill
   - AE activity appeared very sensitive to cylinder pressure ie AE activity would rise & fall as pressure rose & fell

5. AE may not be able to identify/predict immiment burst
   - no AE activity increase was noted at burst; however
     * AE system may have been off-line at burst
     * cylinder had not been recently refilled

6. AET's ability to identify corrosion or cracking mechanisms is unknown (ie recognition of sulphide stress corrosion cracking, crack nucleation, hydrogen embrittlement, fatigue, etc)

7. AET, as tested here, has promise but IS NOT yet market ready

8 Further work, on a large sample of cylinders, is required

9. Further theoretical analysis is required to formulate the minimum set of conditions under which AET can be reliably used
   - temperature, pressure of cylinder & gas
   - past history of cylinder
   - calibration & adjustment of working AET systems
   - impact of cylinder geometry/metallurgy on AET results (diameter, length, wall thickness, hardness, chemistry)

REMAINING QUESTIONS

A number of detailed questions and concerns regarding the use of AET remain unanswered. Among those questions are:

1. Can AET be used during filling instead of immediately after

2. How long is AET data valid/insightful after filling is stopped
   - ie as function of time, temperature, pressure

3. How much variation is there between cylinders
   - nominally identical cylinders
   - different diameters
   - different lengths
   - different wall thicknesses

4. How much AE variation arises from cylinder material properties
   - cylinder temperature
   - material hardness, chemistry
   - cylinder history
     * age
     * stress cycles and reversals
     * stress relieving history

Additionally, a number of questions from or about the specific test data arise, which are tangential in nature but merit attention. Among those are:

5. Is it proper to retire a cylinder if a crack is detected by AET

6. Do cracks self-heal, stop, or run-away; under what conditions
   - if stress corrosion cracking
   - if hydrogen embrittlement
   - if fatigue

7. How do gas temperature, pressure, water content influence SCC

8. How do gas temperature, pressure, water content vary
   - with environmental conditions
   - with distribution line & filling station conditions
   - with time after fill
   - with cylinder pressure before fill began

PRESSURIZED CYLINDER TEST:

The acoustic emission from a CNG fuel cylinder, pressurized with natural gas that contained a wet and sour corrodant was monitored continuously until cylinder failure. The cylinder was identified as "CTC SP1503 3000 85/8046/99 FABER 11-85 CNG ONLY CNG FUEL SYSTEMS CANADA".

Acoustic emission monitoring and fracture results are described below. The test site and mode of failure is discussed in Appendix "B".

PROCEDURE:

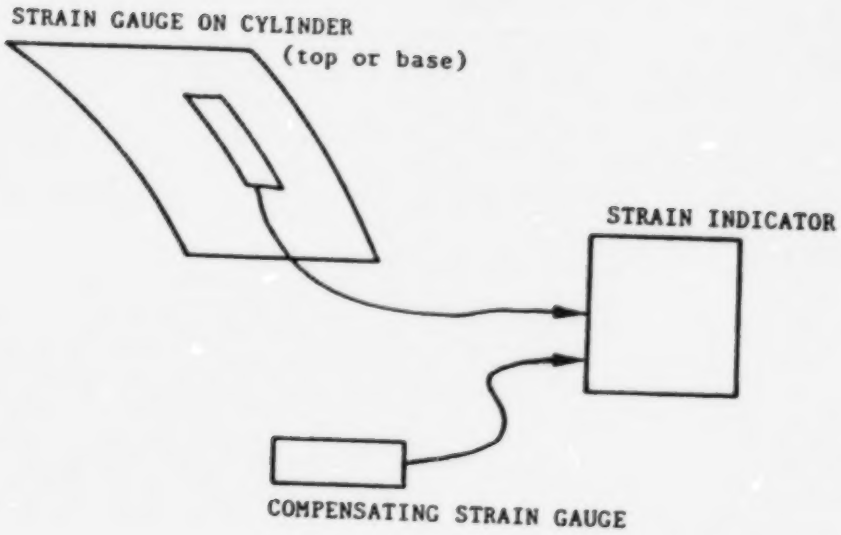
The cylinder was placed in a shallow trench and continuously monitored for acoustic emission. The exposed surfaces of the trench were covered to prevent wind or rain contact with the cylinder, and as described below, a heating blanket was subsequently added to warm the cylinder.

The cylinder position in the trench is shown in Figure 3.1. The cylinder was horizontally oriented with two transducers located at mid-length. A wideband transducer (P.A.C. S/N WD 403) was bonded to the uppermost surface of the cylinder using a cynoacrilate type glue. A second transducer was supported at the 6 o'clock position of the cylinder using a magnet and silicon grease based acoustic couplant. The second transducer was a ~175 KHz resonant frequency transducer. Throughout this report the transducer positions are referred to as top and base respectively.

FIGURE 3.1: The cylinder was positioned horizontally in the trench at a depth of about 0.9 m (3 feet). The wideband transducer (arrow) was located at the uppermost surface (12 o'clock position) and the 175 KHz resonant transducer was located at the 6 o'clock position. Both transducers were positioned near the cylinder mid-section.

The circumferential strain in the cylinder was measured from two foil-type strain gauges, one mounted near each transducer. The strain was measured from each gauge independently and only during the initial cylinder pressurization. Figure 3.2 shows a schematic of the strain measuring apparatus and the results obtained from each gauge.

One litre of an acidified chloride solution (NACE TM-01-77) was placed in the cylinder before attaching the nozzle valve and associated tubing. This solution was not altered throughout the test except by exposure to sour and natural gases.



Cylinder Pressure	MICRO-STRAIN Top	Base
0 psig	0	0
1700	790	720
3100	1440	1210
Stress at 3100 psig	42,000 psi (288 MPa)	35,100 psi (242 MPa)

FIGURE 3.2: Schematic of circumferential strain measurement used during initial cylinder pressurization.

The gas filling system is shown schematically in Figure 3.3. This system was used to both charge the cylinder with hydrogen sulphide and natural gas, and to vent the cylinder. During the test the cylinder was pressurized twice, the second pressurization was intended to refresh the hydrogen sulphide content within the compressed natural gas.

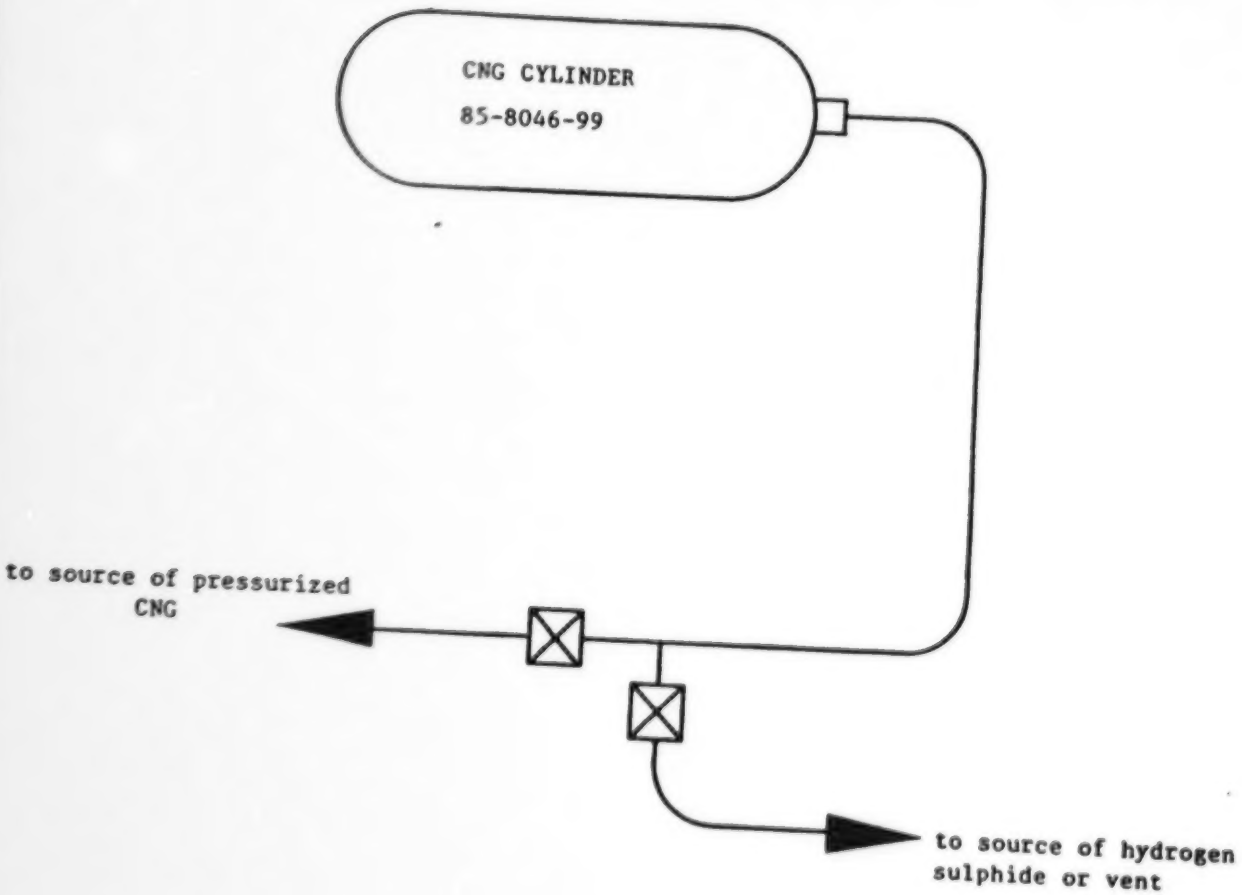


FIGURE 3.3: Schematic of cylinder filling and venting system.

Initial Pressurization:

The cylinder was pressurized with pure hydrogen sulphide gas and vented twice before introducing the initial hydrogen sulphide charge at 40 psig. This procedure was used to reduce the residual air content of the cylinder before pressurizing with natural gas. Natural gas was fed to the cylinder from a mobile bulk compressed nature gas filling vehicle (see Figure 3.4), and the filling of hydrogen sulphide and CNG was controlled using independent high pressure motor actuated valves.

Compressed natural gas was added to a total cylinder pressure of 3100 psig. This pressure was re-established after a 30 minute period allowed for partial thermal equilibration. No further gas was added, and 10 hours after initial filling the pressure was 2600 psig, at an ambient temperature of 5°C. The pressure fluctuated between 2500 psig and 2600 psig dependent upon ambient temperature, and the total exposure period was 145.5 hours.

Second Pressurization:

The cylinder was depressurized to 50 psig and held at this pressure for about 30 hours.

Prior to adding hydrogen sulphide, the cylinder pressure was further reduced to near atmospheric pressure. Hydrogen sulphide was added to a pressure of 90 psig followed by natural gas to a total pressure of 1450 psig. This pressurization signified the commencement of the second pressure cycle at 176 hours from the start of the test. The total cylinder pressure was increased to 2800 psig in three stages:

	Pressure psig	Time
start of repressurization	1450	176 hrs.
second CNG charge	2350	178
third CNG charge	2800	189.5
cylinder rupture	-	190.5

During the second pressurization, the temperature was raised using an electrically heated blanket, and by insulating the area around the cylinder. The temperature was monitored remotely using an ice-water referenced type K thermocouple and throughout the second pressurization the temperature was as follows:

	Cylinder Temperature (°C)	Time
Ambient	1	175 hrs.
Fist CNG Charge	5	176
Second CNG Charge	3.5	187
Third CNG Charge	4.0	189
Cylinder Rupture	5	190.5

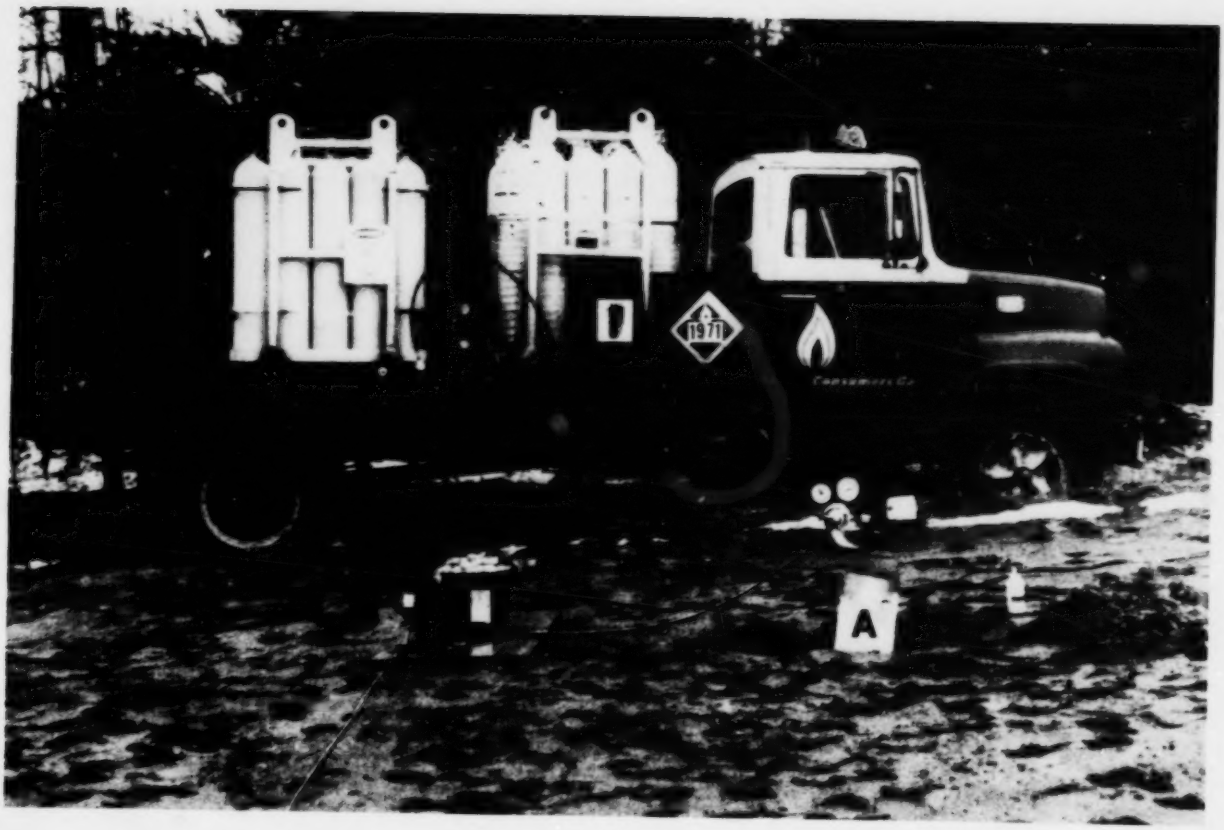


FIGURE 3.4: The cylinder was pressurized using pure hydrogen sulphide (A) and natural gas from a mobile CNG facility. An assembly of two independently controlled valves is shown (B) at the head of the tube leading toward the cylinder.

3.3 ACOUSTIC EMISSION MONITORING:

The acoustic emission monitoring apparatus is shown schematically in Figure 3.5, and the monitoring parameters used are shown in Table 3.1.

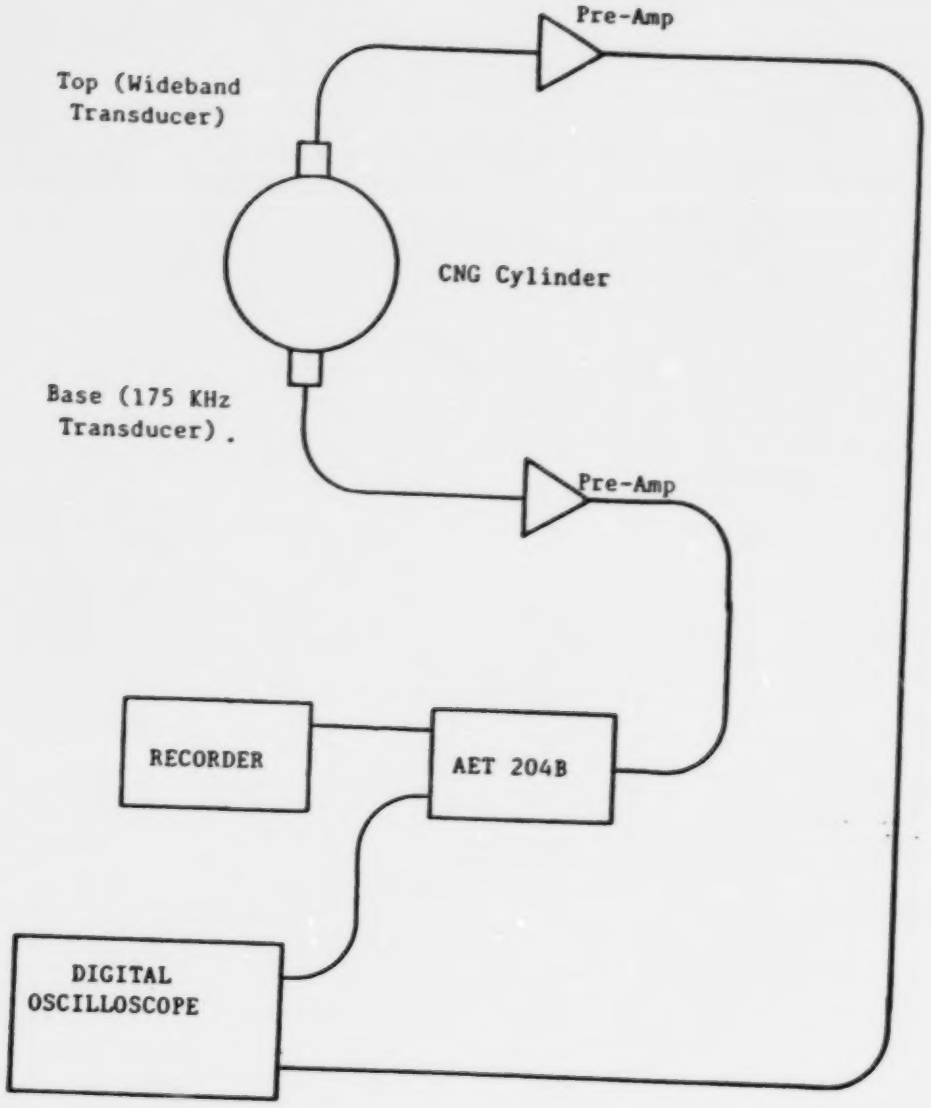


FIGURE 3.5: Schematic of A/E monitoring apparatus.

TABLE 3.1 ACOUSTIC EMISSION MONITORING PARAMETERS USED FOR THE PRESSURIZED CNG TEST CYLINDER.

	Transducer	
	Top	Base
Transducer Characteristic:	Wideband Differential	175 KHz Resonant Differential
PreAmplifier Gain:	48dB	40dB
PreAmplifier Band Pass Filter:	100 KHz to 1 MHz	125 KHz to 1 MHz
Total Signal Amplification:	48dB	70dB
Signal Detection Threshold:		1.00 volt

Recording Parameters:

Oscilloscope:
- Dual Trace digital Recording Oscilloscope (Nicolet Model 2090)
- 20 MHz analog band width
- 50 uS signal sampling resolution

Strip Chart Recorder:
- Chart speed: 2 cm per hour
- 20000 A/E counts full scale deflection

The cumulative acoustic emission counts plotted versus time is shown in Figure 3.6 and 3.7. The general trend observed during the test was that much acoustic emission activity was occurred soon after pressurization, followed by a period of constant activity that appeared to vary with temperature. Very little acoustic activity was observed when the ambient temperature fell to -5°C.

It was significant that the rate of acoustic emission was constant at about 400 counts per hour just prior to cylinder rupture. The cylinder failed rapidly without a notable increase in the A/E rate immediately before rupture.

Typical transducer signals recorded during the test are shown in Figures 3.8 to 3.10. The signals in Figure 3.8 were recorded about 10 hour after the start of the test. These were readily detectable, although low amplitude signals. The signals shown in Figure 3.9 and 3.10 were recorded just prior to cylinder failure. Figure 3.9 shows a signal recorded from the top (wideband) transducer and Figure 3.10 shows a signal recorded from the base transducer.

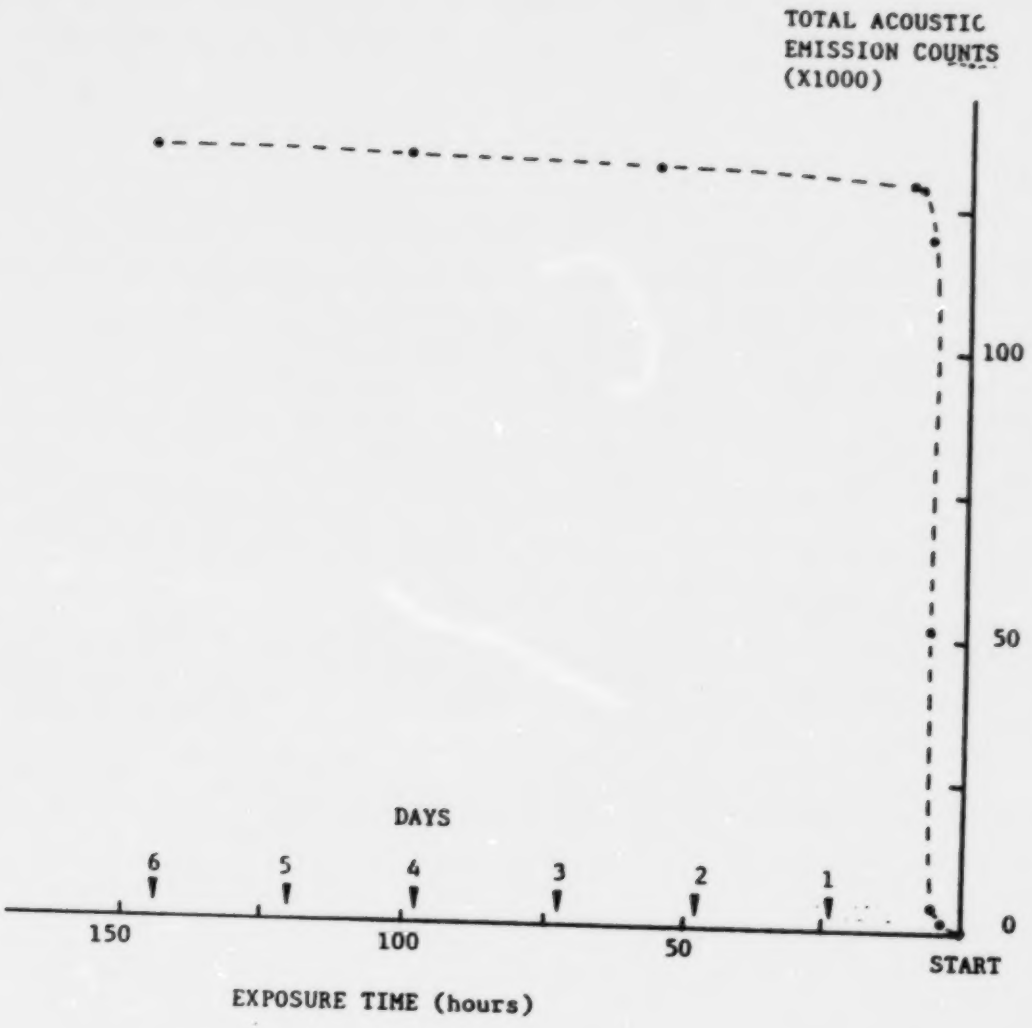


FIGURE 3.6: SN 85-8049-99: Total acoustic emission recorded during the initial pressurization.

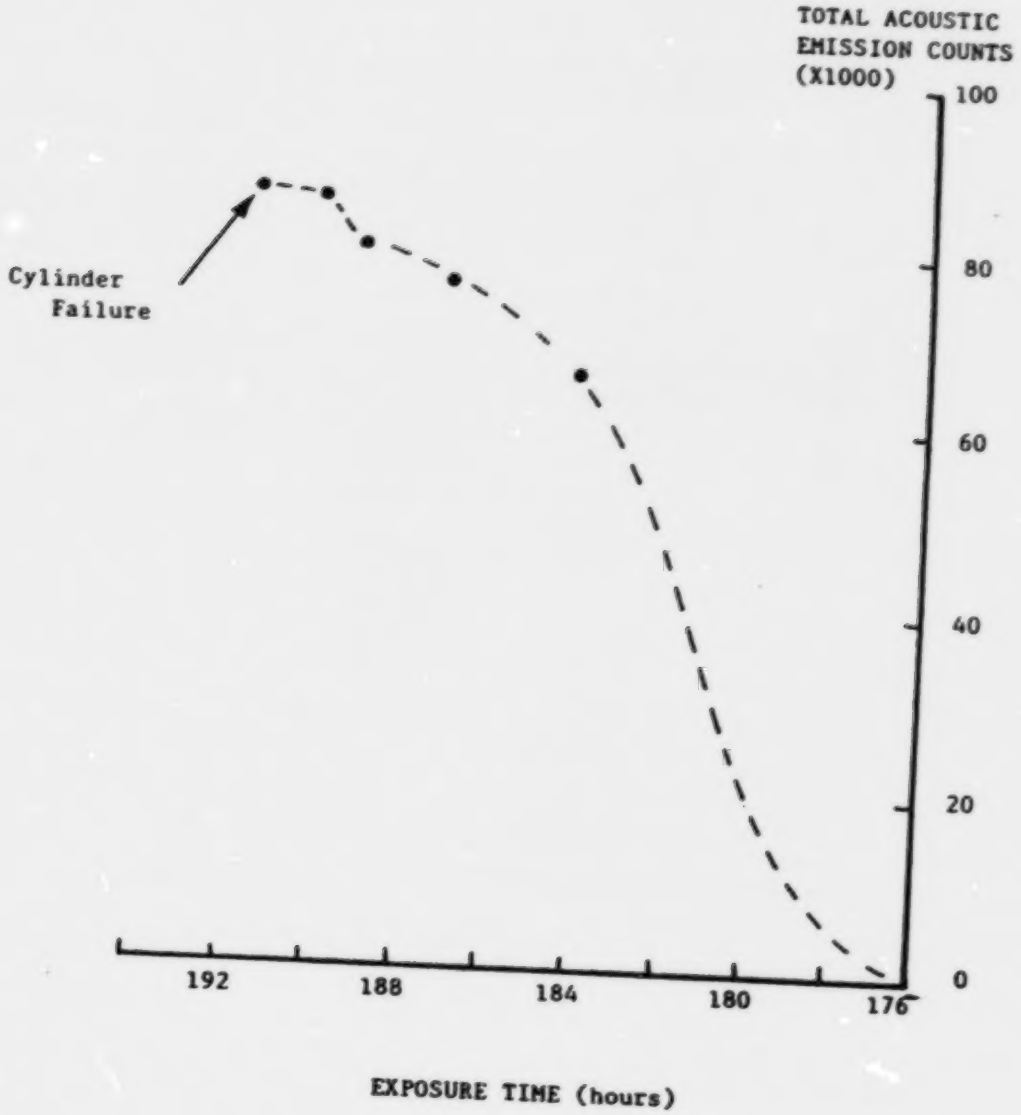


FIGURE 3.7: SN 85-8049-99: Total acoustic emission recorded during the second pressurization.

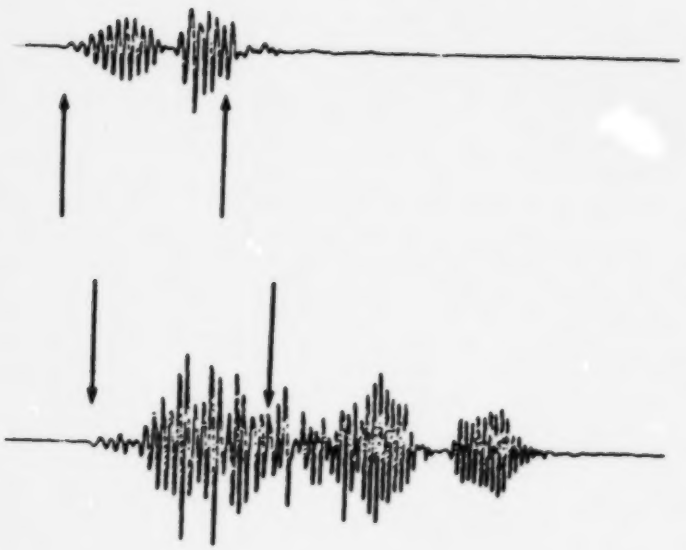


FIGURE 3.8: SN 85-8049-99: Typical signals recorded from the top (wideband) transducer.

Upper Signal Amplitude 37 uVpp (RTI)

Lower Signal Amplitude 60 uVpp (RTI)

FIGURE 3.9: S/N 85-8049-99: This signal was recorded from the top transducer just prior to cylinder failure.

Peak Signal Amplitude: 178 uVpp (RTI)

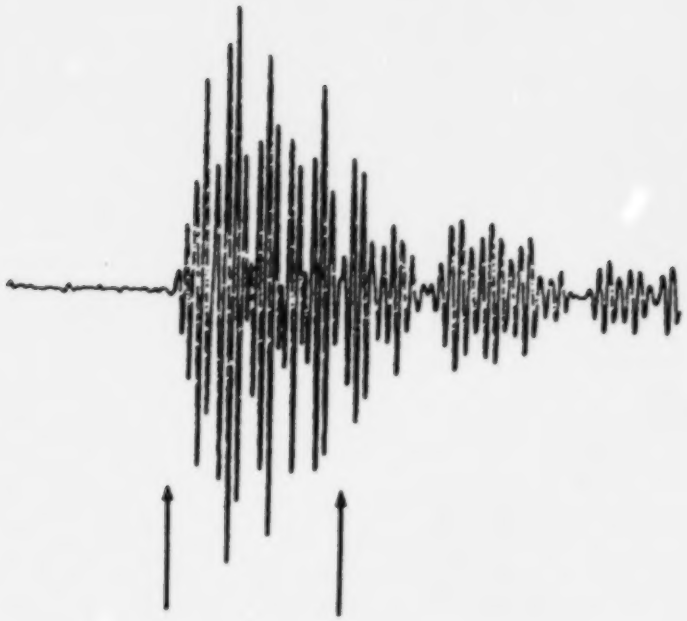


FIGURE 3.10: SN 85-8049-99: This signal was recorded from the base transducer (175 KHz) just prior to cylinder failure.

Peak Signal Amplitude 240 uVpp (RTI)

METALLOGRAPHIC EXAMINATION:

The cylinder failed suddenly resulting in three longitudinally oriented fractures, but only two cylinder fragments. The origin of cracking was along a 4 inch (100 mm) segment of the fracture face at the base (6 o'clock) position of the cylinder. The origin location is shown in Figure 3.11 and the brittle fracture face, typical of stress oriented hydrogen induced cracking (SOHIC) is shown in Figure 3.12

The inner surface of the cylinder was examined using wet fluorescent magnetic particle testing, and as shown in Figure 3.13, additional cracking was observed. The crack indications were longitudinally oriented, and were confined to the area near the fracture origin.

Except for cracking near the cylinder nozzle, the mode of cracking observed on the remaining fracture faces was indicative of ductile shear. The cracking near the cylinder nozzle, shown in Figure 3.14, revealed a fast brittle mode of fracture that may be related to material properties and not due to environmental assisted cracking.

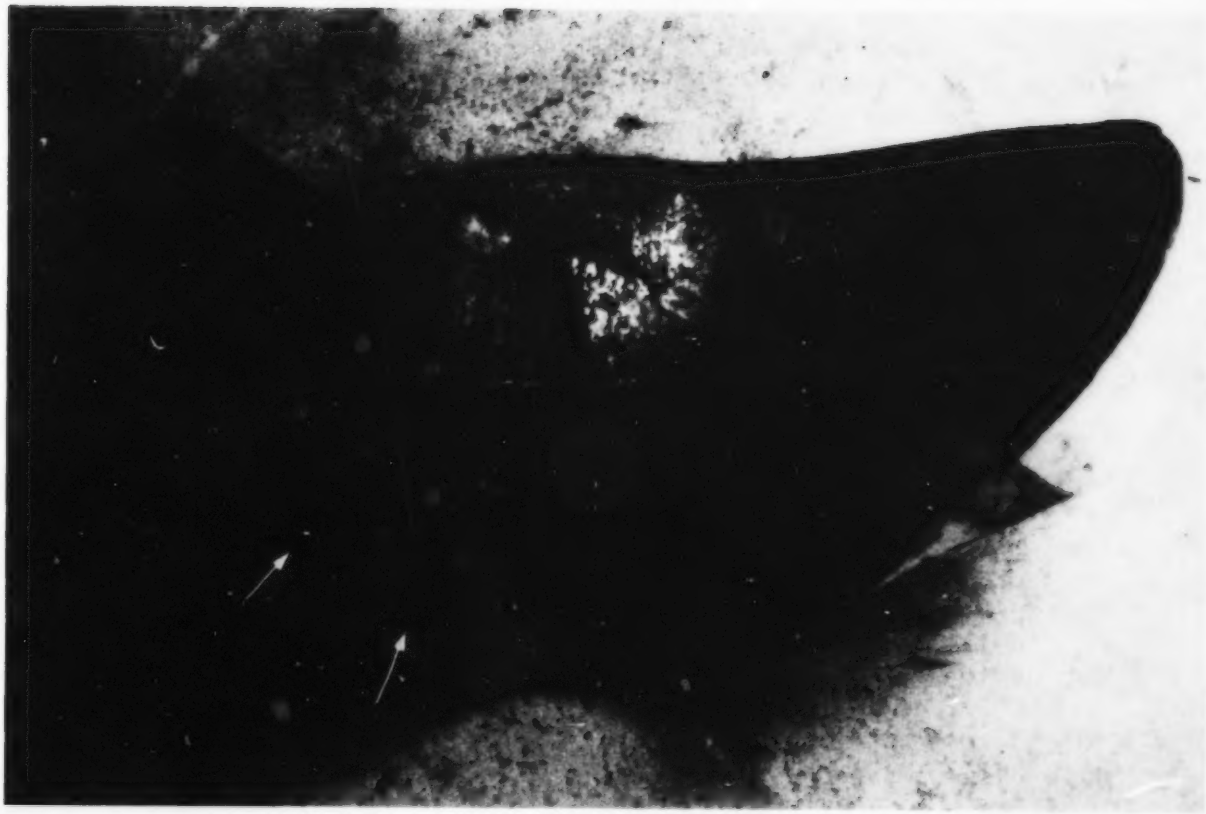


FIGURE 3.11: SN 85-8049-99: The origin of cracking is shown between the arrows.

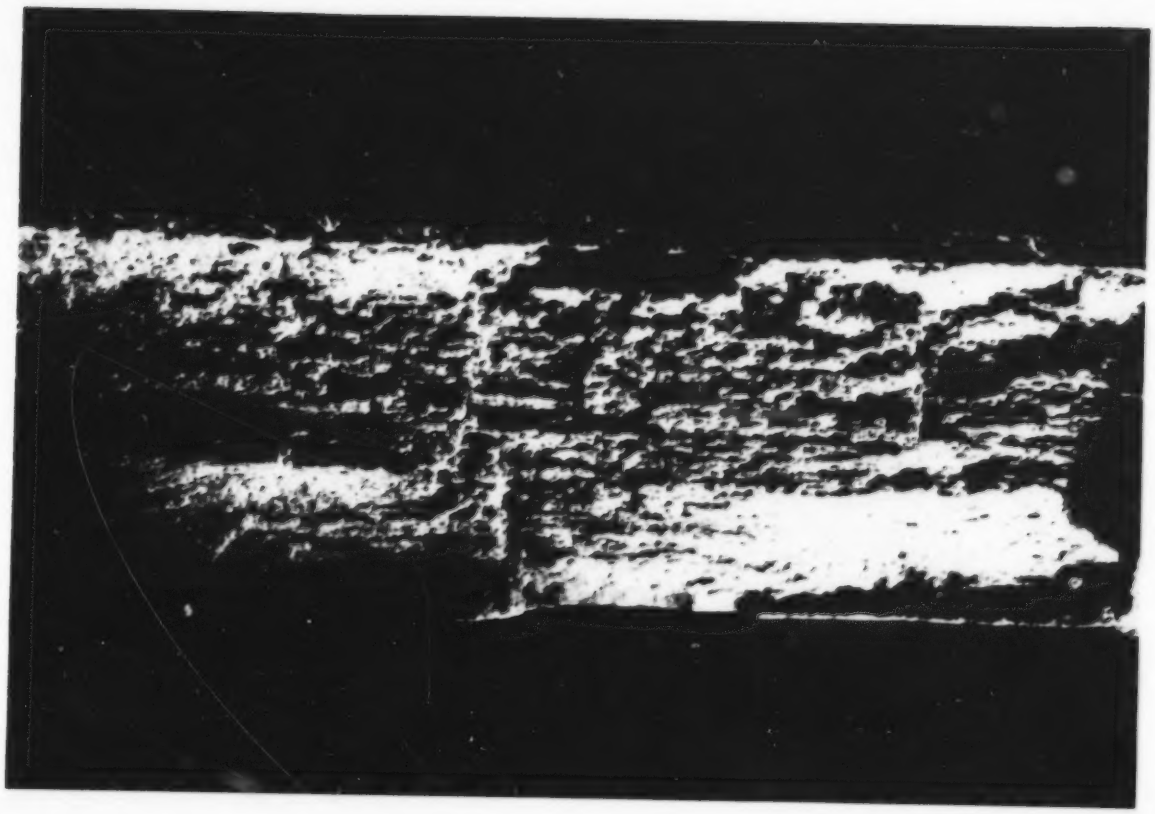


FIGURE 3.12: SN 85-8046-49 (MAG. X4.5): The fracture face at the origin of cracking was characteristic of stress oriented hydrogen induced cracking.

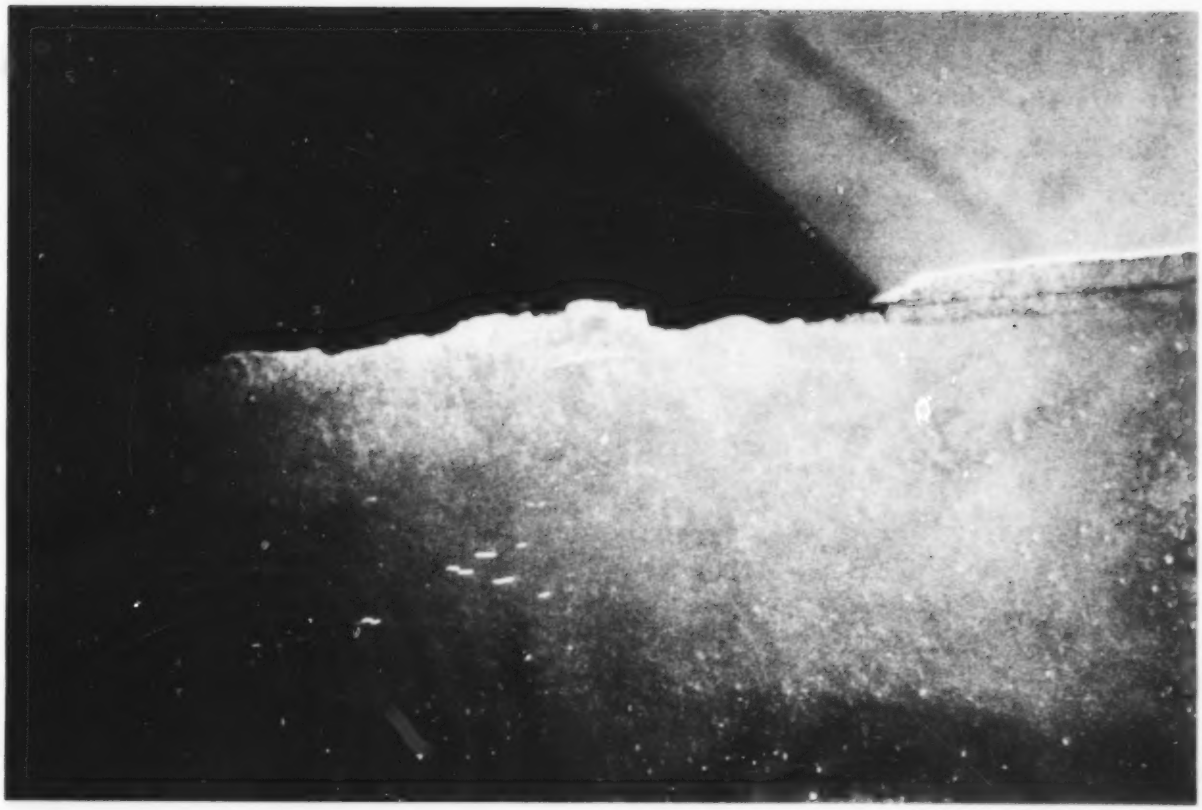


FIGURE 3.13: SN 85-8049-99: Fluorescent photograph showing MT indication on the inner surface of the cylinder near the fracture origin.

FIGURE 3.14: SN 85-8049-99. The crack faces at the nozzle were indicative of fast brittle fracture.

A transverse cross section of the fracture edge was prepared at the origin of cracking, see Figure 3.15. Both through wall and lamellar stepwise cracking (Figure 3.16) were observed near the crack origin.

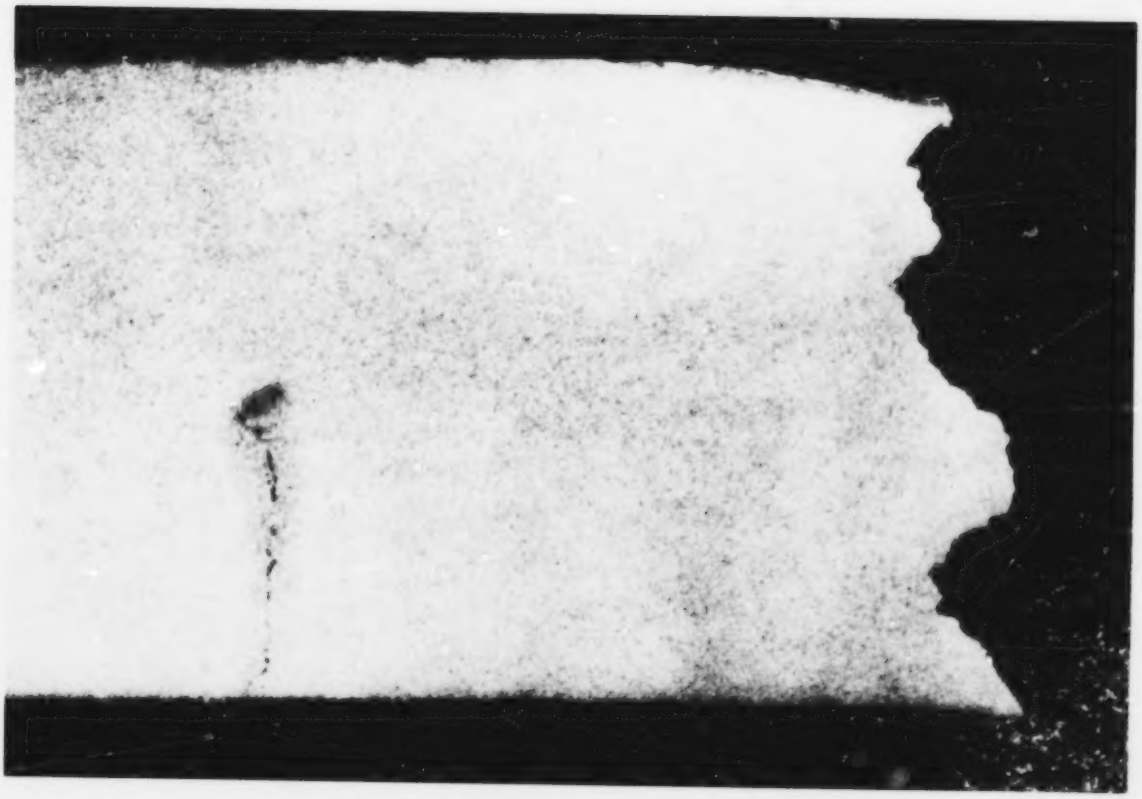


FIGURE 3.15: SN 85-8049-99 (MAG. X8): The crack profile at the fracture origin is shown on the left (the inner cylinder surface is uppermost). Both secondary through wall cracking and lamellar stepwise cracking were observed, see also Figure 3.13. Nital Etch

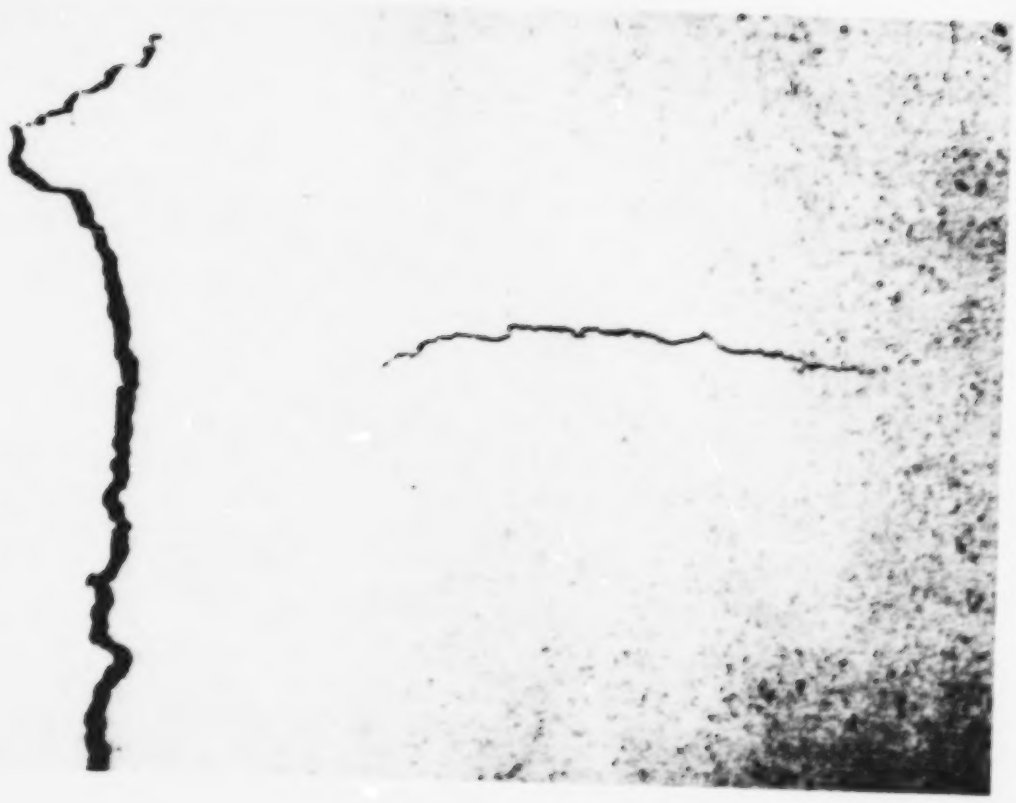


FIGURE 3.16: SN 85-8049-99 (MAG. X25): Lamellar stepwise cracking was found in the cylinder near the fracture origin. Nital Etch

SUMMARY

Acoustic emission monitoring can detect the corrosion or cracking of CNG fuel cylinders exposed to wet sour gas.

The characteristics of the different acoustic emission signals could not be directly related to corrosion or cracking mechanism (crack nucleation, hydrogen embrittlement or sulphide stress corrosion cracking).

A rigorous signal analysis was not carried out on the digitized A/E signals collected to date.

The effect of the corrodant was not well understood. hydrogen sulphide was used to promote cracking in the text cylinders. The cracking occurred only in the area in contact with the acidified chloride solution. Since this was a worse case corroding environment for the CNG fuel cylinder materials, and does not represent a realistic environment to be encountered in CNG service, the effects of the following should be studied:

a) Dry hydrogen sulphide in compressed natural gas;

b) Wet hydrogen sulphide (water only) in compressed natural gas; and

c) Wet hydrogen sulphide and carbon dioxide in compressed natural gas.

The tendency toward corrosion or cracking could be observed by monitoring for acoustic emission, and this data could more reliably be related to the worse case CNG service environment.

Supply and Services Canada / Approvisionnements et Services Canada

Michel Rancourt
Science and Professional
Services Directorate
Supply and Services Canada
11 Laurier Street - 12C1
Hull, Quebec
K1A 0S5

CONTRACT – CONTRAT

REQUISITION NO. – N° DE LA DEMANDE: T8080-6-3115

PAGE 1 OF 1 DE 1 — 8

CONTRACT SERIAL NO. – N° DE SÉRIE DU CONTRAT: T8080-6-3115/01-SZ

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106630-2000-000C-1020-2398

ITEM – ARTICLE	DESCRIPTION – DESCRIPTION	DELIVERY OFFERED – LIVRAISON PROPOSÉE	QUANTITY – QUANTITÉ	UNIT PRICE – PRIX UNITAIRE	TOTAL
1	RESEARCH OF ACOUSTIC EMISSIONS IN NATURAL GAS VEHICLE STORAGE CYLINDERS 31 03 87				

48SZT8080-6-3115

CNG Fuel Systems
2150 Steeles Avenue
Brampton, Ontario
L6T 1A7

COUNTRY OF FOREIGN CONTROL – PAYS DE CONTRÔLE ÉTRANGER: CDN

F.O.B. POINT – POINT F.A.B.: OTTAWA, ONTARIO

VALUE OF FOREIGN CONTROL – VALEUR DES ÉLÉMENTS ÉTRANGERS: $ CDN

GST – TPS: N/A

M. Rancourt

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APPENDIX "B"

PRESSURIZED CYLINDER TEST SITE

and

MODE OF FAILURE

TEST SITE:

A test site was provided at CFB Borden, and as shown in Figure B1 was ideally suited to the project. The CNG fuel cylinder was placed in a shallow trench and covered with a tarpaulin as protection from rain (snow) and wind born debris.

Filling and venting of the cylinder was controlled by a pair of remotely operated motorized valves (see Figure B2). The valves were operable from the A/E monitoring site located in the vehicle. The vehicle (a rented recreational motor home) was positioned behind the concrete bunker shown in Figure B1, and connection to the cylinder required about 100 feet (30 m) of cabling.

The test cylinder was monitored for acoustic emission for 190.5 hours prior to failure. However, after 145.5 hours at pressure, the cylinder was vented and repressurized for the following reasons:

(a) The A/E activity during the first few hours of the test was very high, but after 20 hours, this activity slowly declined to about 30 A/E counts per hour. Without knowing the acoustic emission characteristics that would lead to cylinder failure, the decision to replenish the hydrogen sulphide content was thought prudent.

(b) The weather changed dramatically during the first pressurization. The ambient temperature fell resulting in a reduced cylinder pressure and reduced corrosion reaction kinetics. Prior to repressurization, the cylinder was shrouded with an electric blanket and insulation (see Figure B3) in an effort to stabilize the temperature at about 4°C (40°F).

CYLINDER FAILURE:

The cylinder failed suddenly and without significant increase in acoustic emission activity. Just prior to failure the rate of acoustic emission was 800 A/E counts per hour.

The failure took place about 15 hours after repressurizing the test cylinder with hydrogen sulphide (90 psig) and natural gas. Repressurization was undertaken in three steps, the last of which was to increase the total pressure (using natural gas) to 2800 psig. The cylinder was at this pressure for one hour when failure occurred.

As shown in Figure B4, failure resulted in generally widening the trench and hurtling the cylinder fragments in one direction for about 150 yards (140 meters). The nozzle fitting and tubing were intact and showed little evidence of deformation or damage. This suggests that the cylinder failure did not lead to significant movement prior to splitting the nozzle and releasing the fitting.

The cylinder developed three longitudinal fractures, but parted into two fragments that landed in close proximity to each other, see Figure B5. The impact site of one fragment is shown in Figure B6. It was noted that there were no other impact sites for the cylinder fragments, and that the general concentrec nature of the disturbed sand and snow suggest the fragments dropped to the ground at a high angle.

As described in the text of this report, cracking originated at the 6 o'clock position of the cylinder toward the nozzle end, and propagated rapidly in a longitudinal direction. The crack origin exhibited a brittle fracture morphology about 4 inches (100 mm) long. Except for the tendency toward brittle fracture near the nozzle, the remaining fracture surfaces were indicative of ductile (shear) cracking.

The cylinder fragments are shown in together in Figure B7.

FIGURE B1: The pressurized CNG fuel cylinder was placed in a shallow trench located beneath the tarpaulin (arrow).

FIGURE B2: Motor operated valves were used to control the filling and venting of the test cylinder.

FIGURE B3: The test cylinder was shrouded with an electric blanket and insulation prior to repressurization. Care not to permit contact between the blanket and cylinder was necessary to prevent false sources of acoustic emission.

FIGURE B4: Failure resulted in widening the trench and hurtling the cylinder fragments to the area indicated by an arrow.

FIGURE B5: The cylinder failed into two fragments that fell within close proximity to each other. The arrow indicates the location of the trench in front of the concrete bunker.

FIGURE B6: The impact site associated with this fragment suggests it landed at a high angle (perpendicular) to the ground.

FIGURE B7: SN 85-8046-99: The cylinder contained three principle longitudinal fractures but parted into two fragments.

REFERENCES

1. "Stress Corrosion Cracking and Electrochemical Behavior of Gas Transportation Cylinders in Weak Acidic Media", Vijayan C.P., Industrial Materials Research Institute, N.R.C., Montreal, Quebec.

2. Steels for Gas Containers, Naylor D.J., National Physics Laboratory, Teddington, Middlesex, TW11 0LW, U.K., 1984.

3. "Evaluation of possible corrosion problems in the shipment of compressed natural gas in high strength steel cylinders". Report prepared for SOQUIP, Quebec, 1978.

4. South-West Research Institute, private communication

5. Mr. C. Webster, British Columbia Hydro, Research and Development; private communication.

6. National Association of Corrosion Engineers (N.A.C.E.), Test Method, TM-01-77.

7. "The Role of Hydrogen on Sulphide Cracking of Low Alloy Steels", Herzog E., Corrosion Vol.42, No. 8, Aug. 1986.

8. "On the Kinetics of Environmentally Assisted Fatigue Cracking of Pressure Vessel Steels in the Presence of Hydrogen Gas"; Kesten M., Ronow P.; Metallic Corrosion, 8th International Congress on Metallic Corrosion, Vol. 1, Sept. 1981, Pg. 420 - 425.

9. "Stress Corrosion Cracking of C-Mn Steels in Environments Containing Carbon Monoxide" Parkins R.N., et.al. Materials Performance Vo. 25, No. 10, Oct. 1986, pg. 20 to 27.

10. "Acoustic Emission as a Means of Nondestructive Testing of Compressed Gas Containers", DeMichelis C. et.al.; Metallurgia Italia, Vol. 70, No. 3, March 1978, page 105 - 111.

11. "Corrosion of High-Pressure Steel Gas Containers - a Survey"; Fromberg M., Korrosio, Vol. 15, No. 3, 1984, page 147 to 156.

12. "Hydrogen Induced Stress Corrosion Cracking of Heat Treatable Steels in the Carbon Dioxide - Water System", Kunge E, Becker H., Werkstoffe and Korrosion, Vol. 35, No. 6, 1984, pg. 280 - 281.

13. "Effect of Load Mode on the Hydrogen - Assisted Fracture of an Ultra High - Strength Steel", Takemato M., Corrosion, Vol. 42, No. 10, October 1986, pg. 585 - 592.